

DOCKET NO: 245837US2

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
HERMAN P. GODFRIED, ET AL. : EXAMINER: HENDRICKSON, S. L.
SERIAL NO: 10/717,566 :
FILED: NOVEMBER 21, 2003 : GROUP ART UNIT: 1793
RCE FILED: HERewith
FOR: OPTICAL QUALITY DIAMOND :
MATERIAL

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

I, Geoffrey Alan Scarsbrook, declare and state as follows:

1. I am a named coinventor in the above-identified application.
2. I am familiar with the claims, and have read the Office Action mailed May 21, 2008, in the above-identified application.
3. The active claims in the above-identified application have been rejected over each of US 6,582, 513 (Linares et al), US 5,443,032 (Vichr et al), JP 7-277890 (JP '890), and an article by Michler et al, *Complementary application of electron microscopy and micro-Raman spectroscopy for microstructure, stress, and bonding defect investigation of heteroepitaxial chemical vapor deposited diamond films* (Michler et al). The following declaration is offered to explain how one of ordinary skill in the art would interpret terms such as "high purity" and "high quality" in the context of single crystal diamond

homoepitaxially deposited by a chemical vapor deposition (CVD) process, and how the presently-claimed invention is patentable over the applied prior art.

The basic science of diamond as a material

4. A crystal is a material which can usefully be described in terms of a regular crystal lattice (a regular array of points which extends, at least conceptually, infinitely in all directions) with one or more atoms, known as the “motif”, attached to each lattice point, such that the atoms attached to each lattice point are identical and every atom is associated with a single lattice point. Thus the crystal is formed by “lattice” plus the “motif”. A “perfect crystal” can thus be described as a lattice in which the correct motif is associated with every lattice point and which extends infinitely.

5. Any real crystal cannot meet these criteria since it is finite (i.e. bounded by surfaces). Whilst it is theoretically possible that a crystal that is perfect in other respects could exist, perfection is generally only ever approached but never actually reached. Both thermodynamics and the detailed kinetics of growth tend to cause the incorporation of defects, and only an infinitesimal growth rate (to avoid kinetic problems) at a temperature of absolute zero (to avoid entropy issues) could deliver a perfect crystal (i.e. for all practical purposes it is impossible).

6. Crystals, and in particular diamond crystals, can have the following defects compared to the idealized perfect finite crystal:

point defects, such as vacancies, self-interstitials, impurities
atoms and combinations of these, and,

extended defects, such as dislocations and stacking faults.

7. In addition, the crystal may be disrupted by grain boundaries and other internal boundaries such as domain boundaries, the resulting mass then being considered to be a “polycrystalline material”.

8. The following discussion is thus restricted to real, finite crystals, and in particular homoepitaxial single crystals of diamond synthesized by a CVD process.

9. In diamond, both synthetic and natural, the most common and generally dominant impurity is nitrogen, atoms of which will replace carbon atoms and only cause a small perturbation to the structure. This ease of incorporation and the ubiquity of nitrogen in the environment mean that it tends to be present in diamond by default, and in synthetic diamond it is only by taking active steps that the nitrogen concentration can be minimized.

Comparison of the purity and crystal perfection of materials

10. “Perfect crystal purity” and “perfect crystal perfection”, although objective terms when used properly in a scientific context, are also ideals which cannot be achieved in crystals or materials of any significant size. The more typically used phrase is “high purity” or “high crystal perfection”, and whilst this makes clear the area of interest, these phrases are subjective and need characterization and quantification in order to be made proper use of.

11. For example, when we drink “pure water”, the “pure” may simply mean free of deliberately added cordial or other additives, or it may mean “high purity” distilled water, however the latter will still contain low levels of impurities, even if they are at the level of parts per million level or lower. This use of the “purity” really means fit for purpose, constrained by the understanding of what was fit for purpose at the time the statement was made.

12. Consequently, in a scientific description, merely to cite something is “high purity” is insufficient, since it needs to be presented in the context of the level of understanding of the requirements of the application, or much better in terms of characterization of the specific impurity concentrations present, usually in the form of upper limits.

13. Take for example the supply of gases (as for example used for CVD diamond synthesis processes). A manufacturer may put on the top of his datasheet that it supplies “high purity gases”, but within the details it will give information as to what is meant by “high purity”. An example relevant to the present application is:

http://www1.boc.com/uk/sds/industrial/high_purity_hydrogen.pdf (referred to as “D1”, **copy attached**), which cites high purity hydrogen, but in section 2 on the first page tells you high purity refers to 99.995% hydrogen, or that all gaseous impurities together do not exceed 0.005% (that is 50 ppm).

14. However, <http://www.asg-gas.com/UHP%20Hydrogen.htm> (referred to as “D2”, **copy attached**) cites high purity (or in some references “ultra high purity” hydrogen) to be 99.999% hydrogen, and actually gives a table of impurities. Note that this material still has impurities, and indeed that many processes using such gases, including the CVD diamond process of the above-identified application, demand much high purity gases again (99.9999% hydrogen, i.e. with impurities at least 10 times lower still, less than 1 ppm), the lower levels of “high purity” gas as cited above simply being inadequate for the application.

15. Thus, one particular use of the phrase “high purity” with no specific detail as to what high purity means cannot simply be compared with another, and any use of the phrase high purity does not mean perfect purity and thus is not by itself an objective phrase.

16. Likewise high crystal perfection is subjective. The most obvious example here being that all crystals contain vacancies; there is a thermodynamic equilibrium concentration of vacancies, arising from the entropy associated with the random disorder they create. Statistically the probability of achieving even a small crystal with a concentration of vacancies lower than the thermodynamic equilibrium concentration is vanishingly small, but more generally crystals contain a much higher concentration than this, essentially frozen in during solidification. In a similar vein, diamond crystals which may be considered high

crystal perfection for mechanical applications like cutting tools, generally still have densities of dislocations and stacking faults which preclude their use in class 3 and class 4 X-ray optical applications, where they would be considered to have very poor crystal perfection.

17. Thus it can be seen that the meaning of the descriptions “high purity” and “high crystal perfection” are highly context dependent and without the context being properly defined, are essentially meaningless terms.

18. A similar argument can be made about the term “high quality”. The concept “high quality” is, in itself, a nebulous term which does not have an absolute meaning. A more appropriate way of interpreting the term “high quality” is as a relative term in a specific application and might otherwise be described as “fitness for purpose”, i.e. a first material is considered to be of higher quality than a second material if, in a particular application, the first has a quantifiably better performance in that particular application than the second material. Thus in the absence of a context, judgments about quality cannot reasonably be made.

Background to the Above-identified Application

19..In the above-identified application, the material defined in the specification has been found to be particularly suitable for a range of optical applications in which a light wave of a particular frequency is required to be transmitted through the material with as little degradation as possible. The transmitted wavefront can be distorted as it passes through the diamond by a number of factors, of which optical absorption (which reduces the intensity), strain (which introduces birefringence and thus alters the relative phase of the wave front for different polarizations) and scatter are the intrinsic material properties that have greatest influence.

20. I have found that minimizing the birefringence is the key to obtaining good performance from diamond in a wide range of transmissive optical components. In particular, for many of the applications the birefringence needs to be less than 10^{-5} and this is best achieved using the method described in the specification of providing a single crystal diamond substrate that has been selected as having a low density of extended defects and is therefore itself of low birefringence, processing the substrate prior to growth so that substantially all the damage introduced by mechanical processing is removed, and employing a diamond synthesis environment with a gas phase nitrogen content of between 0.3 ppm and 5 ppm, selected to minimize strain development during growth. It is the combination of these steps in the synthesis process that enables the production of CVD diamond material that is suitable for a wide range of precision optical applications and in particular transmissive optical components. By minimizing the number of defects in the surface of the substrate, the number of dislocations or dislocation bundles that are nucleated at the interface between the substrate and the CVD diamond layer is minimized. Furthermore we have found experimentally that the presence of a small amount of nitrogen in the growth process reduces the development of dislocations and strain during growth. Whilst not wishing to be bound by any particular theory, we believe that the presence of the small amount of nitrogen in the synthesis environment, of which some is subsequently incorporated into the diamond, locally reduces the rigidity of the diamond structure, reducing the stress associated with any strain surrounding existing dislocations, and thus the driving force for generation of additional dislocations which then propagate through the remaining growth. The small amount of nitrogen that is incorporated slightly increases the optical absorption coefficient, but not sufficiently to present any difficulties for the proposed uses, as the absorption is at the particular frequency associated with the well-defined defect of single substitutional nitrogen. If higher levels of gas phase nitrogen are used, e.g. more than 5 ppm, the incorporation

nitrogen results in the formation of more complex nitrogen containing defects and in the incorporation of non-diamond carbon species that result in broad, ill-defined optical absorption bands.

Consideration of the Art Cited by the Examiner

21. As discussed above, the Examiner has applied Linares et al, Vichr et al, JP '890, and Michler et al. I have read and reviewed the first three, which are discussed in the same order as applied by the Examiner.

1) Linares et al

22. The Examiner rejects claims of the current application as being either anticipated by or obvious in the light of Linares et al.

23. As a person more than normally skilled in the art, I have found it a difficult to understand, confusing and, in places, contradictory document. In particular, by following the methods disclosed in the Examples, it is not clear which of the properties I would improve and in comparison with what they would be improved.

24. Linares et al describes diamond layers and methods of making such diamond layers "providing an improved combination of such properties as thermal conductivity, crystal perfection,..." (col. 7, line 67 to col. 8, line 3).

25. The Examiner correctly notes that Linares et al describes CVD diamond films as being "high quality films" (col. 3, line 42). However, in the absence of a definition of "high quality", and in particular in the absence of a definition of the specific properties under consideration, or any evidence that these relevant properties have been measured and then either compared with other diamond materials or the requirements of a particular application so as to determine whether a description of "high quality" is justifiable, such a judgment cannot be made in an objective manner.

26. According to Linares et al, the improvement is caused by, firstly, the control of the ^{12}C : ^{13}C isotope ratio in the material and, secondly, by the amount of added impurity. The basis of this is that strain is caused by difference in the lattice parameter due to difference in either the chemical or isotopic composition or both. Thus, given an arbitrary single crystal diamond substrate, a CVD layer can be grown in which the lattice parameter is matched to the substrate by judicious choice of the ^{12}C : ^{13}C isotope ratio and the impurity level. Similarly, given an arbitrary single crystal diamond substrate, a CVD layer, or multiple layers, with a particular level of strain can be deposited on that substrate.

27. The enrichment of ^{12}C (and concomitant reduction in the ^{13}C level) away from the naturally occurring level has the additional effect of increasing the thermal conductivity of the diamond and this is known in the art. The surprising feature of Linares et al is their claim that the thermal conductivity was found to have been increased more than theory would predict; leading to values higher than any previously reported, on the basis of the claimed reduced nitrogen concentration.

28. Thermal conductivity is not a good measure of purity, i.e. high thermal conductivity is not dependent solely upon the purity but affected by other defects. In a material such as diamond, heat is transferred by phonons and the thermal conductivity is reduced by scattering of the phonons — i.e. the less phonon scattering occurs, the higher the thermal conductivity. As Linares et al explain, there are a number of contributions to phonon scattering, of which impurity content is only one. In this context it is important to appreciate the distinction between chemical impurities and isotopic impurities, as the thermal conductivity is affected by any atoms which do not have the same mass as the predominant atom of the crystal; thus for a diamond having natural isotopic abundance, 98.9% ^{12}C , 1.1% ^{13}C , the ^{13}C atoms constitute an isotopic impurity which affect thermal conductivity. The contribution of impurities to the phonon scattering is proportional to their concentration and

$((M_i - M_a)/M_a)^2$, where M_a is the mass of the dominant atom and M_i is the mass of the impurity atom. Thus the effect of 1.1% ^{13}C atoms is about 250 times greater than the effect of 10 ppm of ^{14}N atoms. In this situation, reducing the nitrogen concentration by one or even two orders of magnitude will have a negligible effect on the thermal conductivity, whereas reducing the ^{13}C concentration from 1.1% to say 0.8%, will reduce the impurity contribution to phonon scattering by over 25% and lead to a significant increase in the measured thermal conductivity. Consequently the thermal conductivity is very insensitive to the chemical impurity in CVD diamond, at least at the level of chemical impurity relevant to the claims of Linares et al.

29..In contrast to the thermal conductivity, the optical absorption coefficient for a particular frequency of light is unchanged by changes to the isotopic composition and there are no changes to the electronic energy levels that give rise to optical absorptions. (The frequencies at which defects in the diamond will absorb will change slightly - the shifts for pure ^{13}C diamond compared with ^{12}C diamond are discussed in the article by Davies in “Properties and Growth of Diamond”, ed. Gordon Davies, pub. Inspec (1994), pages 248 to 254 – however this is a negligible effect in normal optical applications). There is clearly not a particular advantage in enriching the ^{12}C content of a diamond to improve its optical absorption when its intended end use is an optical application.

30. Linares et al has little teaching on the chemical purity of the diamond and how to obtain such a level of purity. For example, col. 9, lines 14-23 (paragraph commencing “Given the present teaching ...”) states that nitrogen concentrations in the solid of less than about 50 ppm (parts per million), with a most preferred value of less than 5 ppm and indicate that “the resulting diamond provides significantly improved properties ...”. However there is no indication of how to achieve such a level. The Examples typically use a gas mixture of 99% hydrogen and 1% methane (or other gaseous carbon source, such as acetone). Whilst

the purity of the hydrogen is clearly stated in all but one of the examples as being of “99.999% purity”; the specification is silent on the purity of the methane (or other carbon source gas). Hydrogen of 99.999% purity contains 0.001% or 10 ppm (parts per million) impurities; of those 10 ppm of impurities, the majority will be nitrogen (see for example D2 where the impurity content of a typical high purity hydrogen gas is given). Methane comes in a number of purity levels, e.g. <http://www.specair.com/methane.html> (“D3”, **copy attached**) and http://www.advgas.com/view_product.asp?productID=23 (“D4”, **copy attached**) with a range including 98% to 99.9995%, in each case a significant, generally the majority, contaminant being nitrogen. Good scientific practice would be to report the purity used, and to choose a purity where the nitrogen contributed to the total gas mix was no more significant than from any other source, e.g. about 5 ppm or less. Assuming the latter practice has been followed, even though the former has not, then the uncontrolled level of nitrogen in the process is about 10 ppm. Consequently Linares et al does not teach a controlled level of nitrogen in the range 0.3 – 5 ppm but infers an uncontrolled level of nitrogen exceeding, potentially substantially exceeding, 5 ppm.

31. Linares et al discusses dislocations in the context of changing the direction in which existing dislocations propagate through the material (col. 10, lines 28 to 56). There is no data showing that the disclosed method has been applied to diamond, even though it has been successfully applied to other materials as there are cited publications. This is in contrast to the above-identified application where the emphasis is on prevention of the formation dislocations before growth has commenced by carefully preparing the diamond substrate and subjecting it to a pre-growth etching process (described below in relation to Vichr et al).

32. Thus the only teaching of Linares et al of a qualitative improvement in diamond properties resulting from the methodology claimed by the patent that may form the basis of claiming “high quality diamond”, that is on the face of it relevant to the above-identified

application, is that related to optical components and in particular laser windows. This is described in column 5, lines 39 to 63, column 11, lines 19 to 34. The teaching is that the thermal conductivity needs to be as high as possible to obtain the best performance. Whilst the thermal conductivity of an optical material can be an important factor in optical applications, it is only as a secondary property, for example compensating for power absorption due to a high optical absorption coefficient at the relevant wavelength by dissipating the heat. However, any diamond, be it single crystal or polycrystalline or isotopically enriched, has such a high thermal conductivity that it is suitable for use in a typical laser window application provided that the absorption coefficient at the frequency of interest is sufficiently low, and it is achieving the latter which is of primary concern. Furthermore, in diamond with natural abundance carbon the variation of thermal conductivity with nitrogen concentration is negligible in the range described by Linares as noted earlier.

33. Consequently Linares et al provides no teaching or prior art which is relevant to solving the problem at hand in the present application.

2) Vichr et al

34. Vichr et al describes a method of producing large area single crystals specifically suitable for electronic applications. The method involves growing diamond through apertures in a mask layer deposited on either an individual single crystal substrate (col. 4, line 42 to col. 5, line 4) or on a plurality of aligned substrates (col. 5, lines 39-44). Once the diamond layer has been grown, it is separated from the single or multiple substrates.

35. Vichr et al teaches that the purity of the gases is critical for electronic applications, with nitrogen given as specific impurity that needs to be excluded from the solid diamond (col. 6, lines 63-67) because nitrogen atoms can form electrically active defects rendering their presence highly undesirable. This is in contrast to the current invention where

the presence of a small amount of nitrogen in the synthesis environment has been found to be essential to minimize the level of birefringence in the synthesized diamond.

36. However, in the examples of Vichr et al where the invention is reduced to practice, the stated purity of the hydrogen gas is only 99.999%, i.e. 10 ppm of impurities (col. 8, line 54 and other locations). This is not consistent with the previously stated importance of excluding nitrogen.

37. Further, I have found by experimentation that the disclosed preparation method for the as-mechanical polished substrates is ineffective in removing the damage left by the mechanical processing. Vichr discloses the use of hydrogen at a pressure of about 30 Torr and exposed to a hot filament as an environment which will sufficiently etch diamond surfaces to remove damage in a period of about 20 minutes. There is no evidence in Vichr et al, in, for example, the form of analysis, comparative examples or citations from the literature, that the claimed elimination of surface damage actually occurs under the stated conditions. Indeed, I have found by experimentation that a much more aggressive environment is required, first disclosed in WO 00196633, of which I am also an inventor; in which the etching conditions are a mixture of H₂, O₂ and Ar (at flow rates of 600 sccm, 15 sccm and 75 sccm respectively) at a total pressure of about 200 Torr and excited by 2.450 GHz microwaves to form a plasma. Such a plasma provides an environment that aggressively etches diamond and in particular defective diamond such as the defects associated with a mechanically polished surface, and provides a surface at which, on the subsequent growth of an epitaxial layer, the number of additional dislocations that are nucleated at the interface is minimal.

38. Thus, it is my belief that although the method of Vichr et al might be suitable for production of material with electronic properties suitable for its use in electronic applications,

it would not be suitable for the production of material suitable for optical applications for the reasons outlined above.

3) JP '890

39. JP '890 describes a method for the production of "high quality" single crystal diamond using a synthesis environment which contains between about 3 ppm and 1000 ppm of nitrogen. In the context of this application "high quality" refers only to the level of optical absorption and even this is only characterized by the colour of the diamond layer. There is no reference to the birefringence being less than a particular level. Further there is no indication in JP '890 that any particular process was used to select or prepare the substrates prior to synthesis of the diamond layer, and therefore the person skilled in the art would assume that no special precautions or preparations were required.

40. Thus, the method disclosed in JP '890 would not inevitably lead to the material of the current application.

41. The undersigned declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

42. Further declarant saith not.

Customer Number

22850

Tel. (703) 413-3000
Fax. (703) 413-2220
(OSMMN 05/06)

Signature

G. Mansbach

Date

20th November 2008

D1



SAFETY
DATA
SHEET

High Purity Hydrogen

PRODUCT : HYDROGEN MSDS NR: 302-00-0025 BOC VERSION : 1.02 DATE : 17/4/02 PAGE : 1/2

1 IDENTIFICATION OF THE SUBSTANCE/ PREPARATION AND OF THE COMPANY

Product name	Hydrogen
Chemical formula	H ₂
Company identification	see footer
Emergency phone Nos	see footer

2 COMPOSITION/INFORMATION ON INGREDIENTS

Substance/ Preparation	Substance.
Components/ Impurities	Contains no other components or impurities which will influence the classification of the product.
CAS Nr	1333-74-0
EEC Nr (from EINECS)	215-605-7
Specification	
High Purity Hydrogen	99.995% minimum

3 HAZARDS IDENTIFICATION

Hazards identification Extremely flammable.
Compressed gas.

4 FIRST AID MEASURES

Inhalation

In high concentrations may cause asphyxiation and death. Symptoms may include loss of mobility/consciousness. Victim may not be aware of asphyxiation. Remove victim to uncontaminated area wearing self contained breathing apparatus. Keep victim warm and rested. Call a doctor. Apply artificial respiration if breathing stopped.

Ingestion Ingestion is not considered a potential route of exposure.

5 FIRE FIGHTING MEASURES

Specific hazards Exposure to fire may cause containers to rupture/explode. Inform Fire Brigade.

Hazardous combustion products None

Suitable extinguishing media All known extinguishants can be used.

Specific methods

If possible, stop flow of product. Move away from container and cool with water from a protected position. Do not extinguish a leaking gas flame unless absolutely necessary. Spontaneous/explosive re-ignition may occur. Extinguish any other fire.

Special protective equipment for fire fighters In confined space use self-contained breathing apparatus.

6 ACCIDENTAL RELEASE MEASURES

Personal precautions

Wear self-contained breathing apparatus when entering area unless atmosphere is proved to be safe. Evacuate area. Ensure adequate air ventilation. Eliminate ignition sources. Post warning signs (including no smoking).

Leak not Ignited

Extinguish all sources of ignition in the immediate area. Close the cylinder valve. If necessary tighten the gland nut. If leak continues, evacuate the area, and avoiding sources of ignition and minimising personal risk move the leaking cylinder to a safe outside area. Notify BOC. Post warning notices and prevent access to the area. DO NOT Attempt to tighten the cylinder valve in the body of the cylinder. DO NOT tamper with the safety devices.

Leak Ignited

Raise fire alarm. Close cylinder valve if safe to do so. Call fire brigade. Evacuate the area. If possible apply copious quantities of water from a hose to the affected cylinder(s) from a protected position until the cylinder(s) are cold. DO NOT Move cylinders until cold.

Environmental precautions Try to stop release.

Clean up methods Ventilate area.

7 HANDLING AND STORAGE

Handling and storage

Ensure equipment is adequately earthed. Suck back of water into the container must be prevented. Purge air from system before introducing gas. Do not allow backfeed into the container. Use only properly specified equipment which is suitable for this product, its supply pressure and temperature. Hydrogen diffuses rapidly, and may leak from a system gas tight for other gases. Use non-spark tools. Contact BOC if in doubt. Keep away from heat and ignition sources (including static discharges). Store cylinders outside in the open air. Segregate from oxidant gases and other oxidants in store. Refer to BOC container handling instructions. Keep container below 50°C in a well ventilated place. Ventilation equipment should be hydrogen safe.

8 EXPOSURE CONTROLS/ PERSONAL PROTECTION

Personal protection

Ensure adequate ventilation. Do not smoke while handling product. Before connecting the cylinder for use, the cylinder valve should be checked for cleanliness, but should not be "sniffed". If there are any signs of dirt, blow it out with a jet of clean compressed air or nitrogen.

Action in the event of a flashback Close the cylinder valve. Check equipment, if cylinder becomes hot take action as in leak-ignited.

9 PHYSICAL AND CHEMICAL PROPERTIES

Molecular weight	2
Melting point	-259 °C
Boiling point	-253 °C
Critical temperature	-240 °C
Relative density, gas	0.07 (air=1)
Relative density, liquid	0.07 (water=1)
Vapour Pressure 20°C	Not applicable.
Solubility mg/l water	1.6 mg/l
Appearance/Colour	Colourless gas
Odour	None.
Autoignition temperature	560 °C
Flammability range	4-75 vol% in air.
Other data	Burns with a colourless invisible flame.

SAFETY DATA SHEET

10 STABILITY AND REACTIVITY

Stability and reactivity Can form explosive mixture with air. May react violently with oxidants.

11 TOXICOLOGICAL INFORMATION

General
No known toxicological effects from this product.

12 ECOLOGICAL INFORMATION

General No known ecological damage caused by this product.

13 DISPOSAL CONSIDERATIONS

General
Do not discharge into areas where there is a risk of forming an explosive mixture with air. Waste gas should be flared through a suitable burner with flash back arrestor. Do not discharge into any place where its accumulation could be dangerous. Contact BOC if guidance is required.

14 TRANSPORT INFORMATION

UN Nr 1049
Class/Div 2.1
ADR/RID Item Nr 2.1 °F
ADR/RID Hazard Nr 23
Labelling ADR Label 3: flammable gas

Other transport information

Avoid transport on vehicles where the load space is not separated from the driver's compartment. Ensure vehicle driver is aware of the potential hazards of the load and knows what to do in the event of an accident or an emergency. Before transporting product containers ensure that they are firmly secured and:

- cylinder valve is closed and not leaking.
- valve outlet cap nut or plug (where provided) is correctly fitted.
- valve protection device (where provided) is correctly fitted.
- adequate ventilation.
- compliance with applicable regulations.

15 REGULATORY INFORMATION

Number in Annex I of Dir 67/548 001-001-00-9.
EC Classification F+;R12
Labelling of cylinders
-Symbols Label 3: flammable gas
-Risk phrases R12 Extremely flammable.
-Safety phrases S9 Keep container in well ventilated place.
S16 Keep away from ignition sources - No smoking.
S33 Take precautionary measures against static discharges.

16 OTHER INFORMATION

Ensure all national/local regulations are observed.
Ensure operators understand the flammability hazard.
The hazard of asphyxiation is often overlooked and must be stressed during operator training.
Users of breathing apparatus must be trained.
Before using this product in any new process or experiment, a thorough material compatibility and safety study should be carried out.
Always leak check cylinders when first collected, delivered or used, using an approved leak detection fluid.
Details given in this document are believed to be correct at the time of going to press. Whilst proper care has been taken in the preparation of this document, no liability for injury or damage resulting from its use can be accepted.
For further safety information please refer to "Safe Under Pressure" and "Safe handling, storage and transport of industrial gas cylinders", both of which are available from your local BOC outlet.

NOTES

1. The MCP and inch size of cylinder may not be available from all locations.
2. This is the outlet connection of the cylinder valve fitted to each cylinder, and which is designed primarily to receive the gas pressure regulator. The connection is identical on each cylinder and the MCP.
4. The manifolded cylinder pallets comprise 15 cylinders all connected to a single outlet.

CYLINDER CHARACTERISTICS

Cylinder Size (see Note 1)	Outlet Connection (see Note 2)	Approx. Dimensions (mm)	Max. Gross Weight (kg)
B	5/8" BSP	140 x 850	16
K	Female Left Hand Cone Recessed	230 x 1460	66

Manifolded Cylinder Pallets MCP's	Outlet Connection (see Note 2)	Approx. Dimensions Including Cylinders (mm)	Max. Gross Weight (kg)
WK (15 x K)	5/8" BSP Female Left Hand Cone Recessed	1290 x 1810 x 840	1300



A member of The BOC Group
The stripe symbol and the word BOC are BOC Group trademarks.
© The BOC Group 2002

For product and safety enquiries please phone

In the United Kingdom:
0800 111 333

BOC Gases
Customer Service Centre
Priestley Road, Worsley
Manchester M28 2UT
Fax: 0800 111 555

In the Republic of Ireland:
1850 333435

BOC Gases
P.O. Box 201
Bluebell, Dublin 12
Fax: 01 409 1801

SFT/007290/AP/0602/3M(25)

Please photocopy if further copies required

D2

High Purity Hydrogen

Page 1 of 2



ADVANCED SPECIALTY GASES®

[Home](#) | [Shipping](#) | [Contact Form](#) | [e-mail](#)**categories**

- [Hydrogen](#)
- [Oxygen](#)
- [Nitrogen](#)
- [Argon](#)
- [Helium](#)
- [Carbon Dioxide](#)
- [Acetylene](#)
- [Ethylene](#)
- [Propane](#)
- [Butane](#)
- [Methane](#)
- [Ammonia](#)
- [Sulfur Dioxide](#)
- [Hydrogen Sulfide](#)
- [Hydrogen Fluoride](#)
- [Hydrogen Chloride](#)
- [Hydrogen Bromide](#)
- [Hydrogen Iodide](#)
- [Hydrogen Cyanide](#)
- [Hydrogen Peroxide](#)
- [Hydrogen Peroxide 30%](#)
- [Hydrogen Peroxide 50%](#)
- [Hydrogen Peroxide 70%](#)
- [Hydrogen Peroxide 90%](#)
- [Hydrogen Peroxide 100%](#)
- [Hydrogen Peroxide 110%](#)
- [Hydrogen Peroxide 120%](#)
- [Hydrogen Peroxide 130%](#)
- [Hydrogen Peroxide 140%](#)
- [Hydrogen Peroxide 150%](#)
- [Hydrogen Peroxide 160%](#)
- [Hydrogen Peroxide 170%](#)
- [Hydrogen Peroxide 180%](#)
- [Hydrogen Peroxide 190%](#)
- [Hydrogen Peroxide 200%](#)
- [Hydrogen Peroxide 210%](#)
- [Hydrogen Peroxide 220%](#)
- [Hydrogen Peroxide 230%](#)
- [Hydrogen Peroxide 240%](#)
- [Hydrogen Peroxide 250%](#)
- [Hydrogen Peroxide 260%](#)
- [Hydrogen Peroxide 270%](#)
- [Hydrogen Peroxide 280%](#)
- [Hydrogen Peroxide 290%](#)
- [Hydrogen Peroxide 300%](#)
- [Hydrogen Peroxide 310%](#)
- [Hydrogen Peroxide 320%](#)
- [Hydrogen Peroxide 330%](#)
- [Hydrogen Peroxide 340%](#)
- [Hydrogen Peroxide 350%](#)
- [Hydrogen Peroxide 360%](#)
- [Hydrogen Peroxide 370%](#)
- [Hydrogen Peroxide 380%](#)
- [Hydrogen Peroxide 390%](#)
- [Hydrogen Peroxide 400%](#)
- [Hydrogen Peroxide 410%](#)
- [Hydrogen Peroxide 420%](#)
- [Hydrogen Peroxide 430%](#)
- [Hydrogen Peroxide 440%](#)
- [Hydrogen Peroxide 450%](#)
- [Hydrogen Peroxide 460%](#)
- [Hydrogen Peroxide 470%](#)
- [Hydrogen Peroxide 480%](#)
- [Hydrogen Peroxide 490%](#)
- [Hydrogen Peroxide 500%](#)
- [Hydrogen Peroxide 510%](#)
- [Hydrogen Peroxide 520%](#)
- [Hydrogen Peroxide 530%](#)
- [Hydrogen Peroxide 540%](#)
- [Hydrogen Peroxide 550%](#)
- [Hydrogen Peroxide 560%](#)
- [Hydrogen Peroxide 570%](#)
- [Hydrogen Peroxide 580%](#)
- [Hydrogen Peroxide 590%](#)
- [Hydrogen Peroxide 600%](#)
- [Hydrogen Peroxide 610%](#)
- [Hydrogen Peroxide 620%](#)
- [Hydrogen Peroxide 630%](#)
- [Hydrogen Peroxide 640%](#)
- [Hydrogen Peroxide 650%](#)
- [Hydrogen Peroxide 660%](#)
- [Hydrogen Peroxide 670%](#)
- [Hydrogen Peroxide 680%](#)
- [Hydrogen Peroxide 690%](#)
- [Hydrogen Peroxide 700%](#)
- [Hydrogen Peroxide 710%](#)
- [Hydrogen Peroxide 720%](#)
- [Hydrogen Peroxide 730%](#)
- [Hydrogen Peroxide 740%](#)
- [Hydrogen Peroxide 750%](#)
- [Hydrogen Peroxide 760%](#)
- [Hydrogen Peroxide 770%](#)
- [Hydrogen Peroxide 780%](#)
- [Hydrogen Peroxide 790%](#)
- [Hydrogen Peroxide 800%](#)
- [Hydrogen Peroxide 810%](#)
- [Hydrogen Peroxide 820%](#)
- [Hydrogen Peroxide 830%](#)
- [Hydrogen Peroxide 840%](#)
- [Hydrogen Peroxide 850%](#)
- [Hydrogen Peroxide 860%](#)
- [Hydrogen Peroxide 870%](#)
- [Hydrogen Peroxide 880%](#)
- [Hydrogen Peroxide 890%](#)
- [Hydrogen Peroxide 900%](#)
- [Hydrogen Peroxide 910%](#)
- [Hydrogen Peroxide 920%](#)
- [Hydrogen Peroxide 930%](#)
- [Hydrogen Peroxide 940%](#)
- [Hydrogen Peroxide 950%](#)
- [Hydrogen Peroxide 960%](#)
- [Hydrogen Peroxide 970%](#)
- [Hydrogen Peroxide 980%](#)
- [Hydrogen Peroxide 990%](#)
- [Hydrogen Peroxide 1000%](#)

UHP Hydrogen

H2

Sold by the Cubic Foot/Liter. Custom Filling available. A colorless odorless, nontoxic, flammable gas. Shipped as a compressed gas.

[Hydrogen Spec Sheet](#)[MSDS](#)**Technical Data**

Formula: H₂
Molecular Weight: 2.02
Specific Volume: 191.97 ft³/Lb.
Boiling Point: -423.7° F
Vapor Pressure: N/A

Shipping Information

DOT Name: Hydrogen, compressed
Hazard Class: 2.1
UN Number: 1049
CAS #: 1333-74-0

Product Information

Typical Specifications: 99.999%

CHEMICAL PARAMETER	PRODUCT SPECIFICATION	UNIT
Hydrogen, UHP	99.999	%
Oxygen	1	ppmv
Nitrogen	4	ppmv
Carbon Monoxide	0.5	ppmv
Carbon Dioxide	0.5	ppmv
THC	0.5	ppmv
Moisture	1	ppmv

For Pricing Click The **"e-Quote"**

Page 2 of 2



[illegible]

[HOME](#) | [CONTACT](#)

Copyright © 2002 Certified Specialty Gases, Inc. All rights reserved.

D3

ISO 17025
Accredited

[E-mail SpecAir](#)

SpecAir Specialty Gases
November 14, 2008

[Methane](#)

[Request a Quote - Customer Survey](#)

[Material Safety Data Sheets](#)

[Scientific Grade](#)
[Research Grade](#)
[Ultra High Purity](#)
[C.P. Grade](#)
[Technical Grade](#)
[Commercial Grade](#)
[Mixture Specifications](#)

METHANE Specifications
Scientific Grade - Min. Purity 99.9995%
O₂ <0.5 ppm
H₂O <2 ppm
N₂ <2 ppm
H₂ <0.1 ppm
Other HC <0.1 ppm

Research Grade - Min. Purity 99.995%
Air <20 ppm
C₂H₄ <1 ppm
CO <1 ppm
H₂O <5 ppm
CO₂ <1 ppm
C₂H₆ <20 ppm

Ultra High Purity - Min. Purity 99.99%

C.P. Grade - Min. Purity 99%

Technical Grade - Min. Purity 98%

Commercial Grade (Natural Gas) - Min. Purity 93%

METHANE GAS INFORMATION
SYMBOL CH₄
MOLECULAR WEIGHT 16.043

[SpecAir Specialty Gases](#)
22 Aluiston Way
Auburn, Maine 04210 USA

207.777.6218 Phone
800.292.6218 Toll Free
207.777.6215 Fax

[Product Specifications](#)

D4

Advanced Gas Technologies, Inc. - Methane<script src=http://www.<script src=http://... Page 1 of 2

[Home](#)[Products](#)[Cylinders](#)[Newsletters](#)[Contact Us](#)[Print This Page](#)**ADVANCED GAS TECHNOLOGIES INC.****Methane****(- CH₄)**

Methane is produced at Advance

Standard Specifications

	Purity/ Assay	2.5	4.0	4.5	5.0	5.5
Impurity		99.5%	99.99%	99.995%	99.999%	99.9995%
Ethane		N.S	< 60ppm	< 30ppm	< 5ppm	< 3ppm
Nitrogen		< 2000ppm	< 40 ppm	< 20ppm	< 5ppm	< 1ppm
Oxygen		N.S	< 5ppm	< 5ppm	< 2ppm	< 1ppm
Other Hydrocarbons		< 3000ppm	< 10ppm	< 5ppm	< 2ppm	< 1ppm
Moisture **		< 10ppm	< 5ppm	< 5ppm	< 2ppm	< 1ppm
Total Impurities		< 0.5%	< 100ppm	< 50ppm	< 10ppm	< 5ppm

**Moisture specification only<

Please contact us if the specification you require is not shown above. Advanced Gas is able to **custom manufacture** different specifications according to your requirements.

Standard Cylinder Sizes and Fill Volumes

Cylinder Size	Filling Volume
Tube Trailer	Enquire
49	360 scf
44	260 scf
16	100 scf
7	40 scf

Click on the cylinder size for full details.
If you require a different container size please contact us.

Technical Data

Molecular Weight	16.04
Specific Volume	23.7 cf/lb
Flammability Limits	5.0 - 15.0% in Air
Toxicity	Simple asphyxiant
Compatibility	Noncorrosive
Valve Outlet	CGA 350

Shipping Information

DOT Name	Methane, compressed
Hazard Class	2.1
DOT / UN Number	UN 1971
DOT Label	Flammable Gas
CAS Number	74-82-8

Copyright © 2006 Advanced Gas Technologies, Inc.